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EG&G ROCKY FLATS

INTEROFFICE CORRESPONDENCE

DATE January 2, 1992

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FROM D.R.Ferrier,Waste Operations,Bldg.750,X6456

SUBJECT HALLIBURTON-NUS TREATABILITY STUDY RESULTS SP/DRF-002-92

Attached is a Treatability Studies Status Report by Shaj Mathew dated December 31, 1991 and excerpts from a Technical Memo detailing some tests which have been completed.

The complete Technical Memo is available at McIntyre for reproduction or further review at the program office.

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JAN 1992
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TO: Don Brenneman

FROM: Shaj Mathew

DATE: December 31, 1991

RE: **TREATABILITY STUDIES STATUS REPORT**

The following report summarizes some of the treatability studies activities currently underway:

1. <u>POND 207C STUDIES</u>

The following salient observations were made on the contents of Pond 207C: contents:

- The salts have an alkalinity (methyl orange) of approximately 300,000 mg/kg of $CaCO_3$. The supernatant liquid has an alkalinity of approximately 150,000 mg/kg of $CaCO_3$. This compares with a value of 61,000 mg/kg that I found in the waste characterization study.

- The salts do not exhibit the characteristic nitrate efflorescence which is predominant in all the surrogates prepared in Duncan even though the nitrate concentration is very high.

- In the process of heating and cooling saturated solutions of the salt in pond water, various phases were seen at various times including: material of a "snow-cone" consistency, thick emulsion, thin emulsion, hard crystals and clear solution. This tallies with observations made during sampling by Jack Templeton.

- The pH of Pond 207C salt/water available for the treatability study is abnormally high (11.25) compared with 10.1 observed during the waste characterization study and 9.5 mentioned in earlier communications.

- It was fairly difficult to remove all the free water when saturated solutions were dried overnight (110°C) for TDS measurements. The water on occasion was trapped under a hard crystalline surface in discrete pockets. In other cases, a mushy slurry remained mushy even after extended periods at 110°C.

<u>Solubility Information</u>

The supernatant solution at room temperature had a solids content of 44.4%. This though, is not representative of the entire salt because only the most easily soluble salts could go into solution. The difference in the crystalline salt and the saturated solution is also evidenced by the fact that the nitrate concentration in the solution was 2.5 times that in the salt.

Lowering the temperature by 5°F did not make any observable solubility differences. However, an experiment is being conducted to measure the solubility at 55°F. The results however are delayed by the fact that the solution is not drying up easily enough for TDS measurements.

Heating the salt/solution at 110° for a certain length of time produced a "creamy emulsion."

Phase Transformation

The phase transformation described earlier, make Pond 207C crystals fairly difficult to characterize. I have observed that the same material at the same temperature can have drastically differing physical natures - from a "pudding" containing 62% solids to a solution having 45% solids. It was also observed that once the emulsion was formed, it was very difficult to dissolve the emulsion, even with excessive dilution.

Among all the methods attempted, the only successful method to get the solids completely dissolved was to lower the pH from 11.2 to 9.2. A considerable amount of acid had to be used to achieve this. This prompted the examination of the alkalinity of the salt/solution which was described earlier.

[illegible] 9.5 – [illegible] 10.7
[illegible] 11.1 –> 11.3

Buffering

Titrations were conducted with solutions of the Pond 207C salt (Figures 1 and 2). As can be seen in both curves, the system buffers twice - once at a pH of about 9.6 and again at a pH of about 6.

2. CEMENT/TRASH TESTS

Studies are being conducted to evaluate the appropriateness of trash addition into the cemented product as is envisioned in the present process concept. The following preliminary observations on the cement/trash formulations appear to indicate that the tests were successful.

- All the cylinders that were cast look good visually.

- All the formulations withstood pressures upto 2000 psi after the 7-day accelerated cure.
- Both freeze/thaw and wet/dry samples look good
- The formulations with trash were more viscous during mixing. This made them more difficult to be deaerated. This is observable as poch-marks on the surface ~~of the cylinders.~~

It has to be noted though that the approach will not be completely proven until the tests run to completion and the TCLP results are in.

3. BELT FILTER STUDIES

The previous status report did not include any hard numbers from the Roediger Belt Press study. The various %solids during the various phases of the study are summarized below:

	Initial % solids on sludge	% solids after gravity drain	% solids after hand belt press
1. 207A (untreated sludge - 250 mls 0.2% Praestol 644BC - 20 mls	5.4	20.4	29.2
2. 207A (chlorinated) sludge - 250 mls 0.2% Praestol 644BC - 40 mls	5.4	29.1	36.0
3. 207B N/C/S (untreated) sludge - 250 mls 0.2% Praestol 644BC - 80 mls	9.5	14.1	21.0
4. 207B N/C/S (chlorinated) sludge- 250 mls 0.2% Praestol 644 BC - 80 mls	9.5	16.0	28.6

4. 30%/60% SOLIDS COMPARISON STUDY

A study was conducted to compare the product resulting from sludges with initial solids concentrations of 30% and 60%. The differing solids contents were obtained by conducting separate tests: one using belt filter and the other using Buchner filtration. The total final volume (after mixing with cement/flyash) of the sludge treated, using the belt filter method, was 25% lower than that obtained using the Buchner fund method. Unfortunately, both the casts, after a 48 hour 90-95° cure, were not strong enough.

The following table summarizes the tests:

BUCHNER FUNNEL TEST

Initial Sludge:
5000 mls having 11.7% solids

Experiment:
To above volume,
Lime 78g
Flyash 448g
Cement 234g was added. It was then subjected to Buchner Filtration under vacuum.

RESULTS

Product volume = 1800 mls

UCS testing:
Withstood pressures up to 5, 12 psi after 2 day 95°F cure

Product composition (calculated)

Dry sludge	17.7%
Water	54.9%
Lime	2.3%
Flyash	13.5%
Cement	7.1%

BELT FILTER PRESS TEST

Initial Sludge:
5000 mls having 11.7% solids

Experiment:
To above volume, 1600 mls of 0.2% Praestol 644BC was added. The water was hand-squeezed out between fields of a belt filter cloth. The cake had 26.2% solids. The cake was mixed with
Lime 75g
Flyash 448g
Cement 236g

RESULTS

Product volume = 1500 mls

UCS testing:
Withstood pressures up to 91, 92 psi after 2 day 95°F cure

Product Composition (calculated)

Dry sludge	25.6%
Water	41.2%
Lime	3.2%
Flyash	19.6%
Cement	10.3%

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PER D.R.FERRIER'S MEMO DATED 1/2/92
EXCERPTS FROM TECHNICAL REPORT
Cover Sheet
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Section 1
Section 4

JAN 1992
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TECHNICAL MEMO

TREATABILITY TESTING PERFORMED AT HALLIBURTON NUS LABORATORY PITTSBURGH, PENNSYLVANIA

FOR

EG&G

ROCKY FLATS PLANT

STABILIZATION PROJECT

DECEMBER 1991

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DEC 30 1991

N. E. T. ROCKY FLATS

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1.0 INTRODUCTION

This document summarizes the treatability study activities conducted at the HALLIBURTON NUS Laboratory located in Pittsburgh, Pennsylvania. These activities were conducted in support of the Rocky Flats stabilization project. The activities conducted consist of the following:

- Polymer Testing
- Chlorination Testing
- Dewatering Tests
- Clarification Testing
- Buchner Funnel Filtration Testing
- Belt Filter Press Testing

Several miscellaneous tests were also conducted in an attempt to improve dewaterability of the sludge.

This document consists of four sections. Section 1.0 is this brief introduction. Section 2.0 describes the testing procedures and provides summaries of the treatability test data. Section 3.0 provides discussions of the results. Section 4.0 provides conclusions based on the results and recommendations for future work.

Appendix A contains a copy of the laboratory logbook.

4.0 CONCLUSIONS AND RECOMMENDATIONS

Based on the results from testing to date, this section will provide conclusions and recommendations for future treatability study work.

4.1 SLUDGE THICKENING

Sludge thickening tests were conducted on the minus 325 mesh material that had been hand-sieved to simulate the particle separation to be accomplished by the hydrocyclone. The minus 325 mesh material was estimated to exit the hydrocyclone at 3 to 5 percent solids, therefore, requiring thickening prior to being recombined with the plus 325 mesh material. Both sizes would be recombined prior to pressure filtration.

After the completion of the testing, a decision was made to eliminate the hydrocyclone since it may not work properly due to the nature of the sludge. As a result of eliminating the hydrocyclone, the thickening test results are no longer applicable to this design. However, the data is useful to provide information pertaining to physical characteristics of the sludge.

The following conclusions summarize the sludge dewatering tests:

- The minus 325 mesh sludge settled at a quicker rate after chlorination with 65 percent calcium hypochlorite.
- The minus 325 mesh sludge flocculated better after chlorination with 65 percent calcium hypochlorite.
- A Betz cationic polymer (3360) was found to be an effective flocculating agent for Ponds 207 A, BN, BC, and BS. The optimum dosage for flocculation ranged between 50 and 100 ppm (2.23 to 4.00 pounds of neat polymer per 1000 pounds of dry solids).
- The percent solids after settling for approximately 4 hours approached the densities in the ponds and ranged from 9 to 21 percent.

- The sludge required a larger surface area for thickening when compared to clarification. The limiting pond for each parameter was 207 BN. The area needed for clarification is 275 ft^2 and for thickening is 11,255 ft^2.

4.2 CHLORINATION TESTS

Oxidation of the sludge with 65 percent calcium hypochlorite was beneficial for sludge handling as well as disinfection of the sludge for pathogens likely to be present from dumping of sewage into the ponds. Of the chlorinating agents tested in the laboratory, calcium hypochlorite is the most practical for disinfecting the sludge. The following conclusions summarize the chlorination testing:

- Calcium hypochlorite (65 percent purity) dosages ranged from 40 to 130 pounds per 1000 pounds of dry solids.
- The addition of calcium hypochlorite to pond water generates a precipitate, which is approximately 3 percent by weight of the water.

4.3 PRESSURE FILTRATION DEWATERING TESTS

The pressure dewatering test results are not significantly affected by the decision to eliminate the hydrocylone; however, this data should be qualified somewhat. The recombination of plus 325 mesh material with minus 325 mesh material was done at a ratio of approximately 1.0 to 0.4, respectively. The sludge contains a larger quantity of minus 325 mesh material compared to the plus 325 mesh material, at a ratio of approximately 4 to 1. The presence of this additional amount of fine-grained material may have adverse effects on the filtration rates. Additionally, the pressure filtration tests were conducted with sludge with an initial percent solids of approximately 35 percent, which must be duplicated by equipment other than the hydrocyclone and the clarifier. The following conclusions summarize the pressure filtration tests:

- The "as is" sludge is not amenable to pressure filtration because the sludge blinds on itself.
- The addition of lime improves the filtration rate of sludges.

- The addition of flyash and cement, following pH adjustment with lime, improves the filtration rate.
- The optimum dewatering formulation included the addition of lime to a pH of 12, followed by the addition of Type C flyash and cement in a 1:1:0.5 ratio of dry solids:flyash:cement.
- The optimum dewatering formulation also successfully achieved the LDR requirements for F006, F007, F009, and the TCLP requirements for the characteristic of toxicity.

4.4 BELT FILTER PRESS TESTING

The belt filter press testing was conducted to answer the question of whether this type of filtration is a viable option for dewatering. The following conclusions summarize the results of belt filter press testing:

- The solar pond sludges can be dewatered effectively with a belt filter press to percent solids ranging from 30 to 35 percent.
- The filter cake from the belt filter press does not appear to be pumpable. This means that the cake must be handled as a solid instead of a slurry.

4.5 RECOMMENDATIONS FOR FUTURE TREATABILITY STUDY WORK

Based on the results of the preliminary treatability study testing to date, and the characterization data summarized in the Waste Characterization Report (HALLIBURTON NUS 1991), recommendations for future treatability work are as follows:

Based on characterization data, Pond 207 C and the Clarifier contents will require oxidation to treat cyanide and possibly tetrachloroethene. These contaminants require treatment so the waste will meet the LDR requirements necessary for acceptance at the Nevada Test Site. The sludge from 207 A and the B series ponds will likely require treatment to disinfect the material, thus killing pathogens as required by the Nevada Test Site (NVO-325, 1991).. The existing data also suggests that sludge handling is improved (i.e., settling and dewatering activities) following oxidation.

It is recommended that the oxidation be pursued with ozone first, because of the ease of generating ozone in the laboratory. If ozone is not successful, then calcium hypochlorite should be tried. If calcium hypochlorite is successful, the dosage can be converted to a dosage for chlorine gas or chlorine dioxide for remediation.

After completion of the oxidation testing, sludge dewatering will require further evaluation. Design parameters for a belt filter press should be determined by a vendor conducting tests in the Pittsburgh laboratory. Concurrently, Larox should return to the laboratory to determine if pressure filtration is compatible with sludge from the belt filter press. Tests evaluating lime, cement, and flyash are still necessary to produce a filter cake at approximately 60 percent solids with the pressure filter. If these materials are still required, then factorial experiments should be conducted to evaluate the appropriate operating range for the successful formula used on the Larox pressure filter. Testing would then continue with a similar approach as described in the Treatability Study Work Plan for durability and LDR requirements.

At the conclusion of the dewatering tests, factorial experiments should begin on the cement formulation. If pressure filtration is not a viable option, cement formation testing should begin at the percent solids obtained by the belt press and should follow Phase I as described in the existing work plan (HALLIBURTON NUS, 1991). The initial center point of the factorial experiment should begin with the formulation developed for the pressure filtration tests. Testing would continue as described in the work plan. It is believed that a successful formulation will be developed quickly based successful results from the TCLP data from the pressure filtration filter cake.

Testing for the clarifier and 207 C will be conducted in a similar fashion as described in the work plan. Input from work conducted at the HALLIBURTON Services Laboratory in Duncan, Oklahoma, may result in some modifications to the scope and possibly reduce requirements for the testing in Pittsburgh, Pennsylvania.